REPORTS

TO THE

LOCAL GOVERNMENT BOARD

ON

PUBLIC HEALTH AND MEDICAL SUBJECTS.

(NEW SERIES No. 46.)

Report to the Local Government Board by Dr. J. M. Hamill on the presence of Calcium Sulphate in Baking Powder and Self-raising Flour.

[*Food Reports, No.* 13.]

Presented to Parliament by Command of His Majesty.

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Dr. J. M. Hamill's Report to the Local Government Board on the presence of Calcium Sulphate in Baking Powder and Self-raising Flour.

Arthur Newsholme,
Medical Officer.
April 3rd, 1911.

The attention of the Local Government Board has recently been drawn to reports by public analysts which have directed attention to the fact that certain baking powders and self-raising flours have been found to contain considerable quantities of calcium sulphate. This substance is not generally considered to form an ingredient of baking powder or self-raising flour, and it appeared that considerable doubt existed as to the significance of its presence, and whether any action by authorities under the Sale of Food and Drugs Acts might be called for in the matter.

Accordingly the Board instructed me to make inquiries with the object of ascertaining the circumstances in which calcium sulphate occurs in baking powder and self-raising flour, and how far, if at all, its presence in these articles may be considered legitimate.

In the course of my inquiries I have visited some of the chief manufacturers of, and wholesale dealers in, baking powder, self-raising flour, and materials for the preparation of these articles in London and the provinces. I have also consulted with public analysts, chemical advisers of baking powder makers and others. I would take this opportunity of expressing my thanks to all who have in different ways assisted me in obtaining information.

For convenience this report is divided into parts as follows:—

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I.—The Nature and Varieties of Baking Powder.

A baking powder is a mixture of finely ground solid materials which on the addition of water and with or without subsequent heating evolves gas. When such a powder is mixed with flour, and water is added, the small bubbles of gas which form throughout the dough give to it porosity and "lightness." Any residue left as a result of this reaction should be odourless, tasteless, and innocuous, and as small in amount as is consistent with the efficacy of the powder.

An invariable constituent of all baking powders is sodium bicarbonate from which carbon dioxide gas is evolved by means of a suitable acidic substance. Ammonium carbonate may also be used as a source of gas in making powders, but it has not found favour amongst baking powder manufacturers in this country, partly on account of its expense and the odour of ammonia which is inseparable from its use. The majority of baking powders used in the United Kingdom have as their acidic constituent tartaric acid or cream of tartar or a mixture of these substances. Acid calcium phosphate is also used in baking powders and, to a limited extent, acid potassium sulphate, but this latter substance is unsuitable on account of its hygroscopic nature and the tendency which it exhibits to char the dough with which it is mixed, producing in this way unsightly black spots. Alum is also capable of acting as an acidic constituent of baking powder, but is no longer openly used in this country for this purpose on account of its generally recognised liability to be deleterious to health.

Baking powders in this country may therefore conveniently be classed into two groups—the "tartaric" powders in which the acidic constituent is tartaric acid, cream of tartar, or a mixture of these; and the "phosphate" powders the acidic constituent of which is acid calcium phosphate. Similarly, self-raising flour is flour with which "tartaric" or "phosphate" preparations are incorporated, along with sodium bicarbonate for the purpose of producing gas when the flour is moistened.*

* This use of phosphates in self-raising flour, properly declared and sold as such, must be distinguished from the addition of phosphates to ordinary flour for the purpose of "improving" its baking qualities—a practice which I have dealt with in a separate report (Food Reports No. 12).

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The observations below relate chiefly to the presence of calcium sulphate in baking powders and self-raising flour; other matters relating to these substances are dealt with only in so far as they bear on this question.

2.—The Possible Sources of Calcium Sulphate in Baking Powders.

Calcium sulphate may find its way into a baking powder as a contamination of one or more of the constituents.

Sodium bicarbonate used in baking powders can be considered as free from calcium sulphate. Baking powder makers find it economical to use only the best grades, and in many cases guarantees are supplied by the maker that his product contains from 98 to 100 per cent. of sodium bicarbonate.

Tartaric acid and cream of tartar may also for practical purposes be considered as free from calcium sulphate. I am informed that amounts as large as one per cent. should never be found in these articles.

Acid calcium phosphate as used in the manufacture of baking powder and self-raising flour always contains calcium sulphate to a greater or less extent according to the method of preparation. In some samples it may not exceed 2 to 3 per cent.; in others it may amount to 50 per cent. or even more of the whole. The reasons for these great variations can best be appreciated by a review of the methods employed in the manufacture of acid calcium phosphate.

3.—Preparation of Acid Calcium Phosphate for use in Baking Powder and Self-Raising Flour.

The basis of "acid phosphate," as it is often called, is usually bone ash. Bones may be collected in the neighbourhood of the factory and calcined at a white heat after the gelatine (size) has been extracted from them. Or bone ash may be imported as such, in which case before further treatment it is calcined at a high temperature since it is important to obtain a product of great whiteness for the purpose of making baking powder. Bone ash obtained in either of these ways contains about 90 per cent. of calcium phosphate.

The next stage consists in treating the bone ash with acid; the details of this treatment vary considerably amongst different makers according to the grade of acid phosphate it is desired to produce. The acid used may be pure phosphoric acid (made by burning phosphorus in air in the presence of water) or commercial phosphoric acid made from bone ash and sulphuric acid, or simply sulphuric acid. Mixtures of phosphoric and sulphuric acids may also be employed.

A manufacturer whom I visited uses pure phosphoric acid made from phosphorus in the preparation of high grade acid calcium phosphate. He also makes a lower grade of acid calcium phosphate, using a mixture of three parts of sulphuric acid and one part of pure phosphoric acid. A still lower quality is made by him with sulphuric acid alone. Another firm in preparing acid calcium phosphate use "commercial" phosphoric acid, which

they make by treating bone ash with dilute sulphuric acid. Equal parts of this commercial phosphoric acid (sp. gr. 1·5) and bone ash are mixed together in leaden vessels, the resulting product being dried on shelves in a current of hot air and then finely ground and sieved. They also make a lower grade of acid phosphate by using equal parts of bone ash and of a mixture consisting of equal parts of sulphuric acid and commercial phosphoric acid made from bone ash. A still lower quality was made at this factory by treating bone ash with an equal weight of sulphuric acid (sp. gr. 134 Twaddell). Another firm makes only two grades of acid phosphate. The better quality is prepared from commercial phosphoric acid and the inferior from sulphuric acid.

Commercial phosphoric acid prepared from bone ash and sulphuric acid commonly contains about 3 per cent. of sulphate, but I am informed that by improved methods it is possible to produce a commercial acid containing as little as 0·5 per cent. of sulphate. It should be noted that bone ash itself may contain an appreciable amount of calcium sulphate. A sample on analysis was found to contain 0·162 per cent. of calcium sulphate and an additional amount of combined sulphuric acid equivalent to 0·62 per cent. of sodium sulphate.

It is obvious that acid calcium phosphate made from commercial phosphoric acid and sulphuric acid may contain very varying amounts of calcium sulphate. When commercial phosphoric acid only is added to the bone ash it is said to be possible to obtain a product containing as little as 2 per cent. or as much as 9 per cent. of calcium sulphate according to the precautions observed in the manufacture. When sulphuric acid alone is used the product may contain as much as 50 per cent. of calcium sulphate. Mixtures of sulphuric and phosphoric acids in varying proportions will of course give values intermediate between these two. Thus an acid phosphate prepared by mixing bone ash with its own weight of an acid mixture containing phosphoric and sulphuric acids in equal parts was found on analysis to contain 33 per cent. of calcium sulphate. Intermediate grades of acid phosphate may also be prepared by mixing acid phosphate made from phosphoric acid with acid phosphate made from sulphuric acid.

Precipitated calcium phosphate obtained in the manufacture of gelatine may be used instead of bone-ash in the preparation of acid phosphate. A certain amount of di-calcium phosphate is present in the precipitated calcium phosphate so that to make "acid phosphate" from this material less acid is required than in the case of bone-ash, and when sulphuric acid is used less sulphate will be present in the resulting product.

Apart from the occurrence of calcium sulphate in acid phosphates as a result of the method of manufacture I am informed that in some cases this mineral is deliberately added to the phosphate as a convenient and cheap diluent, and in such cases the mixture may contain considerably more than 50 per cent. of calcium sulphate.

In the following table, analyses of six samples of "acid phosphate" are set forth; the efficiency or strength (*see* section 4, p. 5)

of each sample as measured by the amount of carbon dioxide liberated from sodium bicarbonate by 0·25 grams of the sample is also given:—

	Sample 1.	Sample 2.	Sample 3.	Sample 4.	Sample 5.	Sample 6.
$Ca H_4 (PO_4)_2$	63·10	48·05	35·51	61·63	47·63	33·85
$Ca_3 (PO_4)_2$	23·25	39·31	11·08	27·68	15·78	4·98
$Ca SO_4$	6·86	4·18	45·75	1·03	30·11	55·75
Matter insoluble in HCl. ...	1·24	0·48	1·20	1·56	1·48	1·36
Moisture	1·52	2·72	4·18	1·18	1·10	2 21
CO_2 in c.c. from 0·25 gm. of sample.	28·15	22·00	15·10	26·70	21·70	15·75

Samples 1, 2, and 3 represent respectively the highest, medium and lowest grades manufactured by one firm; samples 4, 5, and 6 represent similar grades made by another firm.

Sample 1 was prepared by mixing practically equal weights of bone ash and commercial phosphoric acid. Sample 2 was made by mixing bone ash with 60 per cent. of its weight of phosphoric acid. In preparing sample 3 two grades of acid phosphate were used, one made by adding a mixture of approximately equal amounts of sulphuric and phosphoric acids to about 70 per cent. of its weight of bone ash, the other by treating bone ash with about 75 per cent. of its weight of sulphuric acid. Four parts of the former were mixed with nine parts of the latter to form sample 3. Sample 4 is stated to have been made by mixing one part of bone ash with about 1½ parts of phosphoric acid. Sample 6 was prepared by treating one part of bone ash with one part of sulphuric acid (sp. gr. 1·5). Sample 5 consists of equal parts of samples 4 and 6.

In connection with the use of sulphuric acid, especially where the acid has been derived from pyrites, it may be well to refer to the possibility of contamination by arsenic. This is recognised by some makers who have their acid and also their products tested from time to time for the presence of this element.*

4.—The Strength of Acid Calcium Phosphate Preparations.

The "strength" or carbon dioxide displacing power of a "phosphate" preparation varies according to the amount of acid added to the bone ash. Manufacturers usually express the

* The Royal Commission on Arsenical Poisoning, in their Final Report, 1903 (Cd. 1848), recommended that no substance used in food, whether intended for consumption alone, or mixed with other substances, should contain more than $\frac{1}{100}$ grain of arsenic per pound if solid, or per gallon if liquid. Special mention is made of the occurrence of arsenic in baking powders in Appendix 24 to the Final Report of the Commission (Cd. 1869). The limit of the Commission should always be observed in judging the suitability of phosphates or baking powders for purposes of food preparation.

The occurrence of arsenic in tartaric acid and cream of tartar is dealt with in the report to the Local Government Board by Dr. A. W. J. MacFadden on "Lead and Arsenic in Tartaric Acid, Citric Acid, and Cream of Tartar." Reports of Inspector of Foods. No. 2. 1907.

strength of their product in terms of cream of tartar; thus a "phosphate" is described as being as strong as, or 15, 20, or 30 per cent. stronger than cream of tartar. Some firms aim at sending out a preparation equal in strength to cream of tartar; others aim at greater strength. One firm makes an acid phosphate from bone ash and phosphoric acid which is 30 per cent. stronger than cream of tartar and another preparation from sulphuric acid which is equal in strength to cream of tartar.

The methods by which the strength of acid phosphate used for baking powder is determined vary amongst different makers. A rough method consists in comparing the amounts of sodium bicarbonate necessary to neutralise equal quantities of cream of tartar and acid phosphate respectively, the neutral point being ascertained by means of an indicator. Or, different quantities of a solution of sodium bicarbonate are evaporated down with the acid phosphate and the residue tested with hydrochloric acid for effervescence. Another method consists in measuring the amount of carbon dioxide liberated from sodium bicarbonate by the acid phosphate in question. For example one maker aims at producing a "phosphate" of such strength that half an ounce of it will liberate 18 cubic inches of carbon dioxide from an excess of sodium bicarbonate.

5.—Formulæ for Compounding "Phosphate" Baking Powders.

Manufacturers are enabled from a knowledge of the neutralising power of their "acid phosphates" to mix the active ingredients in proportions most suitable for the production of an efficient baking powder. In actual practice it is impossible to attain such exactness in mixing the sodium bicarbonate and "phosphate" as to ensure perfect neutrality after interaction; the final product is practically always acid or alkaline to some extent. A maker informed me that he endeavoured to avoid a final alkaline reaction since an unpleasant colour and flavour may develop as a result of the action of the alkali on the flour. Actually, however, a baking powder prepared by this maker was found to contain more sodium bicarbonate than was necessary for the neutralisation of the phosphate present. A large number of other baking powders which have been examined were also found to contain sodium bicarbonate in excess.

For the preparation of baking powder the highest grade acid phosphate, *i.e.*, that containing the least amount of calcium sulphate is generally preferred; low grade powders are said to give a bitter taste to the article in which they are used (*see* p. 9).

In order to keep the acid phosphate and sodium bicarbonate from too intimate contact a neutral non-hygroscopic powder is added, such as corn-flour, rice-flour or a mixture of these. A powder of this kind is usually known as "filling." Rice-flour is generally preferred to corn-flour for this purpose since the latter is said at times to transmit an unpleasant flavour to the article in which the baking powder is used. The amount of "filling" which enters into the composition of a baking powder varies greatly. In high class "phosphate" baking powders the

"filling" commonly forms about one-half of the whole, but in cheaper powders the amount of filling used may be considerably greater than this.

Baking powder is generally retailed in small quantities such as penny packets, and by adding more "filling" it is possible to increase the size of the packet—a consideration which appeals irresistibly to many purchasers—without incurring much extra expense and without in any way increasing the strength of the powder.

The following are typical recipes for the preparation of baking powder recommended by different firms dealing in acid phosphates:—

(1)—50 parts of acid phosphate.
25 „ sodium bicarbonate.
25 „ maize starch or rice flour.

(2)—37 parts of acid phosphate.
23 „ sodium bicarbonate.
40 „ rice flour.

(3)— 2 parts of acid phosphate.
1 „ sodium bicarbonate.
3 to 10 „ rice flour (according to grade required).

(4)—77 parts of acid phosphate.
41 „ sodium bicarbonate.
50 to 100 „ ground rice.

From ½ to 1 ounce of baking powder, according to its strength, is the amount usually required for one pound of flour.

A baking powder prepared according to formula (1) from acid phosphate containing 50 per cent. of calcium sulphate and used in the proportion of only ½ an ounce to 1 pound of flour would contribute over 50 grains of calcium sulphate to the flour.

In preparing a baking powder for sale careful and uniform mixing of the ingredients is important, and the materials used and the boxes in which the powder is packed should be thoroughly dried. Baking powder should be stored in a dry place as otherwise it rapidly loses its efficiency especially when "acid phosphate" is one of the ingredients.

6.—A Comparison between "Phosphate" Baking Powder and ordinary ("Tartaric") Baking Powder.

From the trade point of view the chief advantage which a "phosphate" baking powder possesses over a "tartaric" baking powder is cheapness. Acid calcium phosphate can be obtained from 12 shillings to 30 shillings or more per cwt. according to grade. This enables a baking powder to be produced at about half the cost of a "tartaric" powder of equal strength. Further, it is claimed that owing to the slowness with which gas is evolved from a "phosphate" baking powder in the cold, dough in which it has been used may be left standing for some time before cooking without appreciable loss of "lightness." On the other hand one of the disadvantages under which a "phosphate" baking powder labours is the rapidity with which it loses strength on account of the hygroscopic nature of the acid phosphate used; one maker informed me that with ordinary care it would not keep more than a month. Certain makers claim that "phosphate"

baking powder keeps as well as the "tartaric" variety, but this is not the experience of the majority.

"Phosphate" baking powders are not commonly found in retail establishments probably on account of their indifferent keeping qualities. They are used chiefly by confectioners, bakers, and others who make their baking powder from the acid phosphate as they require it, all risk of deterioration being in this way eliminated. Manufacturers of acid phosphate may make baking powder for sale, but their chief trade consists in supplying wholesale dealers, self-raising flour makers, bakers, and others with acid phosphate for aërating flour or making baking powder according to a formula or prescription supplied with the phosphate. In this connection it should be remarked that acid calcium phosphate is frequently sold under fancy names which suggest an affinity to cream of tartar or tartaric acid. This practice, which is misleading and especially undesirable in view of the difference in price of the two articles, ought to be discontinued. Acid phosphate may, however, be considered as a cream of tartar substitute, and no objection could be taken to its being sold as such on its merits, provided the difference between the two substances is made clear.

7.—Self-Raising Flour.

Self-raising flour is simply flour to which the active constituents of baking powder have been added in the proper proportions. Most self-raising flours are made with sodium bicarbonate and acid calcium phosphate which has practically superseded tartaric acid or cream of tartar for this purpose. The quantities recommended for use vary slightly amongst different makers, but 6 lbs. of "phosphate" and 3 lbs. of sodium bicarbonate to 280 lbs. of flour represents a fairly usual mixture.

The lower grades of acid phosphate, *i.e.*, those containing relatively large quantities of calcium sulphate are generally used in making self-raising flour. Although these lower grade phosphates are not so strong as the higher qualities their inferiority in strength is more than counterbalanced by their cheapness. If the acid phosphate used in preparing self-raising flour according to the above formula contained 50 per cent. of calcium sulphate, the self-raising flour thus produced would contain as much as 70 grains of calcium sulphate per pound.

Self-raising flour is in many cases sold at the same price as ordinary flour and this is rendered commercially possible by using a lower grade, and therefore less expensive, flour in making the self-raising article.

8.—A consideration of the arguments adduced by Manufacturers and others in defence of the presence of Calcium Sulphate in Acid Calcium Phosphate used for Baking Powder and Self-Raising Flour.

It is asserted by certain manufacturers that, owing to the conditions under which the trade is carried on at present, the manufacture of a low grade acid phosphate containing much calcium sulphate has become a commercial necessity in order to meet the

demand which exists or has been created for a low-priced article. It is also maintained that calcium sulphate serves a useful purpose as "filling," and in this way materially increases the stability of the finished baking powder. A further reason adduced in favour of the use of an acid phosphate rich in calcium sulphate is that it greatly improves the appearance of the articles made from the flour in which it is present; in the words of a maker of self-raising flour, it is impossible to obtain the same "clean clear results" from a powder practically free from calcium sulphate as from one containing a considerable amount of this substance. It is also asserted that low grade acid phosphates, notwithstanding the large amounts of calcium sulphate they contain, are not much inferior in strength to grades containing very small amounts of sulphate, and this coupled with the great difference in cost between the two grades offers every inducement to use the lower grades in preference to the higher. It is further stated that during cooking the calcium sulphate disappears and is not to be found as such in the finished article. Finally it is claimed that calcium sulphate is an important ingredient of baking powder since it acts therein as an acidic constituent, in this way replacing acid phosphate and forming a substitute which enables considerable economies to be effected. These arguments are briefly considered below.

In view of the statement that calcium sulphate is able to act as an acidic constituent in "phosphate" baking powder it was thought desirable to investigate this point. Accordingly some experiments were made in this connection by Mr. C. H. Cribb, B.Sc., F.I.C., the results of which are given in Addendum A, p. 14. It appears that under certain conditions calcium sulphate is able to facilitate the liberation of carbon dioxide from sodium bicarbonate with the formation of sodium sulphate and calcium carbonate, but that the amount of carbon dioxide so liberated is not greater than that which can be liberated by heat alone. Calcium sulphate also appears capable of reinforcing the action of acid calcium phosphate, but this reaction is appreciable only when the solution of phosphate and sodium bicarbonate is maintained at the boiling point. Since, however, the temperature of the centre of a loaf reaches 100° C. only for a brief period, the conditions which obtain would appear to be such as would render this reaction negligible from the point of view of practical baking. Furthermore in actual baking experiments with a baking powder containing calcium sulphate, 75 per cent. of the calcium sulphate was recovered unchanged from the finished loaf.* Other experiments would seem to indicate that the calcium sulphate which has disappeared as such in the loaf is again reformed by the agency of the acid in the gastric juice when the bread is eaten.

One manufacturer who was aware of the formation of sodium sulphate at the expense of the calcium sulphate was of the opinion that this interaction was detrimental since, on cooking, the amount of sodium sulphate which may be formed is some-

* Mr. R. A. Cripps, F.I.C., has found in similar baking experiments that on the average 50 per cent. of the calcium sulphate remains unaltered—*see* Annual Report of the Public Analyst for the County Borough of Bournemouth, 1908.

times sufficient to impart a distinctly bitter and unpleasant flavour to the article in which the baking powder was used.

The assertion that low grade phosphates are little inferior in strength to high grade phosphates requires consideration. In this connection and as an illustration of what follows reference may be made to the table on p. 5. The strength or efficiency of a phosphate powder varies with the amount of soluble acid phosphate which it contains. The amount of soluble acid phosphate and calcium sulphate in a powder depends on the amount and kind of acid used in its preparation. If commercial phosphoric acid is employed the amount of soluble acid phosphate depends on the amount of acid added and the calcium sulphate content of the powder depends on the proportion of sulphate present in the acid and also on the amount of acid used. If sulphuric acid is used the amount of soluble acid phosphate and sulphate produced depends up to a limit on the amount of acid employed, but in this case the proportion of soluble acid phosphate which it is possible to obtain is not so great as can be obtained by the use of phosphoric acid. This is due to the fact that when sulphuric acid alone is used part of the calcium phosphate (bone ash) is appropriated for the production of calcium sulphate and phosphoric acid, by which the remainder is transformed into soluble acid phosphate, whereas when phosphoric acid alone is used the whole of the calcium phosphate (bone ash) is available for transformation into soluble acid phosphate. An " acid phosphate," therefore, properly made with an adequate amount of phosphoric acid has a higher soluble acid phosphate content and a higher efficiency than a powder prepared from sulphuric acid. The only support to the contention of the manufacturer is that if insufficient phosphoric acid is used the content of soluble acid phosphate and therefore the efficiency of the powder may only be equal to, or even actually lower than, a powder prepared from sulphuric acid and containing an amount of calcium sulphate many times greater than the powder prepared from phosphoric acid.

If the statement is true that the presence of calcium sulphate is desired because it is capable of improving the appearance of the articles made from the flour with which it is mixed, the presence of this substance in self-raising flour cannot be considered desirable from the point of view of the consumer, since the " improvement" would ordinarily mean that an inferior grade of flour was supplied in place of flour of a better class.

To permit the presence of calcium sulphate in a baking powder on the ground that it serves as a " filling " is a course which cannot be justified. Such a concession would only lead to the general addition of calcium sulphate to baking powder, a procedure which would be to the interests neither of the consumer nor of the more reputable manufacturers of this article.

It may be urged by those who are in the habit of using impure acid-phosphates that the presence of calcium sulphate in foods containing these phosphates is immaterial from the point of view of the consumer, as there is no reason to consider it harmful. The same plea has been made in other cases, for example, when talc is added to rice. But quantities of foreign matter (calcium sulphate) which at first sight seems trifling—for example $\frac{1}{4}$ or $\frac{1}{2}$ per

cent. in cakes, scones, etc., made with baking powder or self-raising flour—becomes material when the large and universal consumption of those articles is considered. It is true that no direct experimental evidence exists that calcium sulphate in small quantities exerts any definitely prejudicial effect upon health, yet the addition in appreciable quantities of needless mineral matter which contributes nothing to the value of the baking powder and may impose an extra and unnecessary burden upon the excretory mechanism is to be deprecated.

I gathered that many manufacturers regret that trade circumstances should have so operated as to have encouraged the production of an acid phosphate containing a large amount of calcium sulphate. They recognise the undesirability of the presence of a large proportion of foreign and useless mineral matter in an article purporting to be a baking powder and would welcome a restriction being placed on the amount of calcium sulphate allowed in an acid phosphate used in the preparation of baking powder and self-raising flour. Chemical manufacturers would have no difficulty in keeping the calcium sulphate in phosphates used for food purposes below a specified limit provided this were not too stringent. It is not difficult to produce an acid calcium phosphate containing less than 5 per cent. of calcium sulphate; and after discussion with manufacturers I am of opinion that a maximum limit of 10 per cent. of calcium sulphate would for all practical purposes give them an ample margin.

In these circumstances it seems desirable that the principle should be adopted that acid calcium phosphate used for the preparation of baking powder and self-raising flour should not contain more than 10 per cent. of calcium sulphate. In baking powder or self-raising flour containing x per cent. of acid calcium phosphate this would mean a maximum limit of $\frac{x}{10}$ per cent. of calcium sulphate. Thus in the case of a baking powder containing 30 per cent. of acid calcium phosphate the maximum limit for the calcium sulphate content would be 3 per cent.; for self-raising flour containing 2 per cent. of acid phosphate the limit would be 0·2 per cent. of calcium sulphate.*

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*In this connection the variable composition of "acid phosphate" should be noted. For this reason in analysing a baking-powder or self-raising flour with a view to ascertaining the proportion of calcium sulphate originally present in the "phosphate," the determination of the $SO_3 : P_2O_5$ ratio in the mixture would seem to have advantages. Assuming that a "phosphate" contained the whole of the phosphoric acid in the form of calcium phosphate $Ca_3(PO_4)_2$ ten per cent. of calcium sulphate in such a phosphate would give an $SO_3 : P_2O_5$ ratio of 1 : 7. In a commercial acid phosphate containing ten per cent. of calcium sulphate the percentage of P_2O_5 would, however, be greater and the $SO_3 : P_2O_5$ ratio would be, say, 1 : 8 or more. In any case in which for practical purposes a ratio of 1 : 7 is adopted the margin of error would be on the side of the maker of the product. In the case of self-raising flour due allowance should be made for phosphorus naturally present in the flour; sulphates occur in only the merest traces in the ash of flour. Loss of phosphorus is liable to occur if incineration is carried out in the ordinary way. Neumann's method or one of the modifications thereof for determining phosphorus by "wet ashing," is free from this disadvantage; *see*:—

A. Neumann. Zeitschrift für physiologische Chemie Vol. 37, p. 129, 1902.

R. H. A. Plimmer and W. M. Bayliss. Journal of Physiology. Vol. 33, p. 439, 1906.

9.—General Conclusions and Recommendations.

Acid calcium phosphate of the lowest commercial quality containing large proportions of calcium sulphate as an impurity is used to a considerable extent by makers of self-raising flour, confectioners, wholesale cake-makers, &c. Less commonly this low grade phosphate is an ingredient of baking powders sold as such to the public. Where the maker concerned buys his acid phosphate as such, and selects the lowest grade as the cheapest, he may obtain a slight additional profit from its employment, which, however, may be counterbalanced by the lower efficiency of the article. It does not appear that the consumer derives any benefit: on the contrary he is liable to be prejudiced by the use of this material, for the reasons given above.

If acid phosphates of relatively low commercial quality and purity are used in food preparation, the interests of the majority of the traders concerned, as well as those of the public, would be served by the existence of an understanding as to the degree of impurity which should entail their rejection as food ingredients. Apart from any administrative action which may be recommended to public authorities on this matter, I have reason to believe that recommendations, as an outcome of my inquiry, would now be welcomed by many traders as a basis for action by them, and the following are made with this object:—

(*a.*) *Manufacturers of acid phosphates* should not prepare even their cheapest qualities of acid phosphate, for sale as a food ingredient, in such a way that it contains more than 10 per cent. of calcium sulphate, and should be in position to guarantee that this figure is not exceeded. Phosphate preparations should not be sold under names which may lead the purchaser to believe that he is buying preparations of tartaric acid or cream of tartar.

(*b.*) *Bakers, self-raising flour makers,* and others using acid phosphates in the preparation of food, should either limit themselves to acid phosphate of high commercial quality, containing a negligible amount of calcium sulphate and pure in other respects, or, if they use lower grades, should in all cases stipulate that calcium sulphate is not to exceed 10 per cent.

(*c.*) *Vendors of baking powders* containing acid phosphates should stipulate that the powders do not contain any higher proportion of calcium sulphate than would be contributed by a commercial acid phosphate containing 10 per cent. of calcium sulphate. They should not receive or sell such baking powders under descriptions which may lead the purchaser to believe that he is buying preparations of tartaric acid or cream of tartar.

Action by local authorities under Sale of Food and Drugs Acts.—In view of the considerations set out above, it would seem desirable that local authorities under the Sale of Food and Drugs Acts should from time to time invite reports from the public analyst as to the proportions of calcium sulphate in a series of samples taken in the district, of (*a*) baking powders which appear to contain acid phosphate of lime, (*b*) acid phosphates supplied to confectioners, &c., (*c*) self-raising flours. Attention could in this way be drawn to those articles which showed a conspicuous

excess of this impurity, and where necessary the institution of proceedings under Section 6 of the Sale of Food and Drugs Act, 1875, should be considered.

J. M. HAMILL.

ADDENDUM.

REPORT BY MR. C. H. CRIBB, B.Sc., F.I.C., ON THE ALLEGED UTILITY OF CALCIUM SULPHATE IN "PHOSPHATE" BAKING POWDERS.

It is claimed that calcium sulphate when present in calcium phosphate baking powders is capable of playing the part of an acid, and so can replace with advantage a portion of the acid phosphate usually employed.

The reactions which have been suggested as justifying this claim are two in number, (1) a direct one between the sulphate and the bicarbonate, and (2) an indirect one between the sulphate and one of the bye-products of the action of the acid phosphate on the bicarbonate.

(1)—*The Direct Reaction.*

When calcium sulphate is mixed with sodium bicarbonate in the presence of water carbonic acid gas is evolved according to the following equation:—

$$CaSO_4 + 2NaHCO_3 = CaCO_3 + CO_2 + H_2O + Na_2SO_4.$$

The evolution of gas commences immediately, but is very slow, so that at the end of half an hour only 59 per cent. and after one hour 79 per cent. of the theoretical quantity was found to have been liberated, and even after three hours the reaction was not complete.

If sodium carbonate is substituted for the bicarbonate a similar change takes place, but no carbonic acid is evolved.

It is obvious therefore that the action of the sulphate is not equivalent to that of an acid and that the maximum amount of carbon-dioxide that can be liberated in this way cannot exceed the proportion which could be driven off from the bicarbonate by the action of heat alone.

In endeavouring to decide whether this reaction is of any benefit to the baker it is necessary to bear in mind the two stages through which the dough or sponge has to pass:—

(*a*) the *cold* period during which the flour, water, and baking powder are being mixed, including also any further time after mixing, during which the dough is allowed to stand before being baked, and

(*b*) the *hot* period during which the baking takes place.

Since as much carbon-dioxide can be liberated from sodium bicarbonate by heat alone as by calcium sulphate it is plain that during this hot period nothing is to be gained from the latter, by the reaction under consideration.

During the cold period the matter is more complicated. Immediately the water, flour, and powder are mixed gas commences to be evolved, but as the acid phosphate acts rapidly on the bicarbonate while the calcium sulphate acts only slowly no increase in the available gas worth considering is obtained unless some time is allowed to elapse betwen the mixing and the commencement of baking.

Furthermore in practical baking other conditions would rarely be so favourable as to admit of this reaction proceeding to any appreciable extent.

(2)—*The Indirect Reaction.*

When acid calcium phosphate acts upon sodium bicarbonate one of the products of the reaction is hydrogen disodium phosphate thus:—

$CaH_4(PO_4)_2 + 2NaHCO_3 = Na_2HPO_4 + CaHPO_4 + 2CO_2 + 2H_2O$,

and this salt in turn reacts under suitable conditions with calcium sulphate giving rise to phosphoric acid which in turn can liberate a fresh quantity of carbon dioxide from any carbonate which may be present. The first part of the reaction may probably be represented as follows:—

$3Na_2HPO_4 + 3CaSO_4 = Ca_3(PO_4)_2 + 3Na_2SO_4 + H_3PO_4$.

Hydrogen disodium phosphate, itself alkaline to litmus, when mixed with calcium sulphate in the presence of water becomes faintly acid after prolonged standing in the cold and strongly so when boiled, the extent of the acidity depending on the time the boiling is continued. When a carbonate is present carbonic acid is liberated, the amount again depending on the time of boiling. In mixtures of acid calcium phosphate, calcium sulphate, sodium carbonate, and water after all direct action between the acid salt and the carbonate has ceased, further quantities of gas are evolved when the mixture is heated to the boiling point. Actual ebullition seems to be an essential condition if good results are to be obtained as at temperatures only slightly below 100° C. the action is extremely slow and uncertain. Thus in a laboratory experiment the acidity developed by the reaction after the mixture had stood in the cold for 15 minutes and had been kept at 98° C. for half an hour, only amounted to one ninth of the theoretical quantity.

Now it has been shown (Catlin, J. Analyt. Chem. vol. 4) that the temperature at the centre of a loaf during baking does not reach the boiling point of water for more than one minute at the most and there is every probability that this degree of heat is often never reached at all. Whether that is so or not the utility of the reaction is very largely discounted by the high temperature needed for its occurrence. To get anything like a substantial yield of gas from this reaction without prolonged boiling it was found to be necessary for the baking powder to contain a proportion of calcium sulphate greatly in excess of the theoretical quantity, *i.e.*, it must be altogether overloaded with that substance.

The presence of much calcium sulphate in the residue left by the powder after it has done its work in the loaf seems to be unavoidable. The statement that it is converted into the easily soluble sodium sulphate is not borne out by experiment. A portion of a loaf baked with a powder containing pure acid calcium phosphate, calcium sulphate, and sodium bicarbonate far in excess of the neutralising power of the phosphate, was dried, very finely powdered, and shaken up with chloroform. A heavy sediment of mineral matter was obtained which contained no calcium carbonate, and in which only one quarter of the total combined sulphuric acid was present as sodium sulphate.

To whatever extent the formation of sodium sulphate may occur during baking, the ultimate fate of the combined sulphuric acid is to be reconverted once more into calcium sulphate when it reaches the stomach. A mixture of tri-calcium phosphate, sodium sulphate and excess of dilute hydrochloric acid of the strength of the gastric juice, after standing for a short time was found to contain a residue insoluble in strong acid and consisting, as would be expected, entirely of calcium sulphate.

CECIL H. CRIBB.

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